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GB 1509098
GB 1493567
GB 1418719
GB 1383407
GB 1379186
GB 1369352
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EP A2 0096566

(58) Field of search
C5D
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(54) Powder detergent of high density

(57) A powder detergent composition having a high density and water-solubility comprises an organic component a part or all of which forms a continuous phase and particles of an inorganic component such as an alkali which are dispersed in the continuous organic phase. The inorganic component may be used to neutralise the organic component.

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Fig.1

Fig.2.

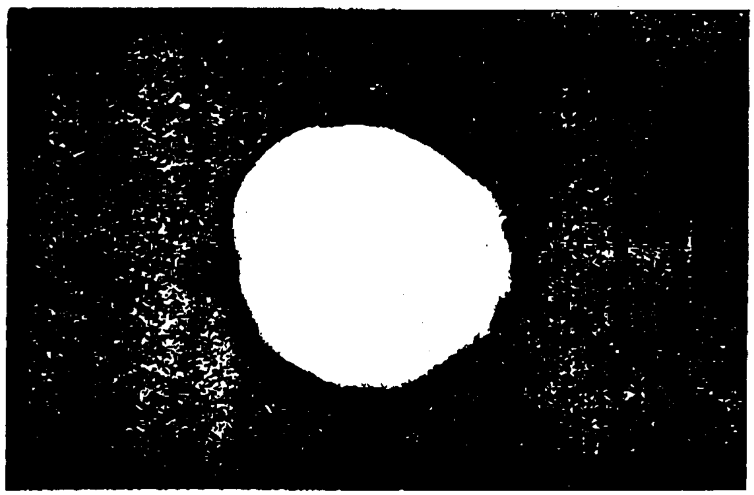


Fig.3.

SPECIFICATION

Powder detergent of high density

The invention relates to a high density powder detergent composition and a process for preparation of the same. It offers improved dispersibility and solubility in water on use. It is also free flowing.

Most household powdered detergents are prepared by spray drying in a low-density form, in view of advantages that mass production is possible, and that the solubility of the product in water is high.

Recently, a high-density powdered detergent has been desired, because it would be advantageous in view of the economy of energy, its transport and in that a housewife can easily carry it.

Attempts to enhance the density of a powdered detergent, have been made for example, Japanese Patent Laid-Open No. 61511/1973 discloses a process for preparing a detergent, having a bulk density of 0.5 g/cm^3 or above, which comprises mixing a surfactant with a builder, in the form of an aqueous slurry, drying the resulting mixture with a drum drier, extruding the dried mixture and granulating the extrudate with a granulator. Further, Japanese Patent Laid-Open No. 36508/1978 discloses a process for preparing a detergent, having a bulk density of 0.55 g/cm^3 or above, which comprises mixing a powdery surfactant with a builder by a dry process.

Hitherto, powdered detergent has been produced either by a spray drying process or dry blending process. Furthermore, anionic and nonionic surface active agents have usually been used as the detergent active substances.

Some smaller manufacturers, of detergent or cleaning agents, employ a dry blending process which requires no large investment cost, as compared with that in the spraying drying process, and they mainly use a small amount of nonionic surface active agents as the detergent active ingredient.

Foaming, during laundru, is considered important, in view of the customs of laundry in Japan, and requires the blending of an anionic surface active agent. However, it has been difficult in the dry blending process to blend the anionic surface active agent in excess of several percent of the total detergent weight because of the adhesive and caking nature of detergent products. Accordingly, large detergent manufacturers prepare an aqueous slurry containing an inorganic builder and an anionic surface active agent, which is then spray dried to obtain the detergent product.

However, resource- and energy-saving have been keenly sought in recent years, and the bulkiness of the previous detergents provides a problem in transportation of the product as well as in domestic use.

In view of the energy saving possible, several processes for producing detergents containing anionic surface active agent without spray drying have already been proposed. For example, Japanese Publication No. 30962/1977 discloses a process for producing a powdered high-density detergent in which a fatty acid is neutralized with a hydrated powdered sodium carbonate at a temperature higher than its melting point. In this process, however, heating is required, the solubilizing power of the obtained detergent is insufficient and cleaning ability is poor because it is based on soap. Furthermore, US Patent Specification No. 3434974 discloses a process of neutralizing an alkyl benzene sulfonic acid with sodium hydrogen carbonate, sodium tripolyphosphoric acid or the like. However, since sodium hydrogen carbonate is used and a relatively great amount of water is involved in this process, the shape of the detergent obtained is restricted only to that of a bar.

Furthermore, US Patent Specification No. 3597361 discloses a process of atomizing an alkyl benzene sulfonic acid and a concentrated liquid of sodium hydroxide, in a rotating drum charged with powder containing sodium tripolyphosphate as a builder. However, this process has a large drawback in that zeolite can not be used as the builder.

However, it is known that high-density powdered detergents obtained by the above processes do not generally exhibit a sufficiently high dissolution rate, presumably because the porosity, which is critical for the dissolution rate, is lost by enhancing the density. On the contrary, the low-density powdered detergent obtained by spray drying has a high porosity and a high solubility, but has the disadvantage that particles of the detergent tend to disintegrate, and the fine powder generated is a nasal irritant,

Summary of the invention

The inventors of the present invention have experimented to provide a high-density powdered detergent having a high dispersibility and solubility in water, and have found that a high-density powdered detergent, having a high dispersibility and solubility in water can be obtained independant of its porosity, by forming a continuous phase which comprises an organic component of the detergent as a matrix. The present invention is based on this finding.

The present invention provides a high-density powdered detergent, characterized in that part or the whole of an organic component constituting the detergent form a continuous phase and that particles of an inorganic component constituting the detergent are dispersed and enclosed in the continuous phase.

In this specification, "high-density" refers to a bulk density of 0.5 g/cm^3 or above.

In other words, a powder detergent composed according to this invention has a high density improved solubility and comprises an organic component, a part or all of which forms a continuous phase, and particles of an inorganic component which are dispersed in the continuous organic component.

An anionic surfactant is an essential ingredient of the organic component, which forms the continuous

phase, or matrix, of the powdered detergent, according to the present invention. Examples of the anionic surfactant include the following compounds.

1) Alkylbenzenesulfonates wherein the alkyl group, is a straight-or branched-chain and has an average number of carbon atoms of between 10 and 16.

2) Alkyl or alkenyl ether sulfates having 0.5 to 8 moles per mole of ethylene oxide, propylene oxide, butylene oxide, ethylene oxide/propylene oxide in a ratio of between 0.1:9.9 and 9.9:0.1 or ethylene oxide/butylene oxide in a ratio of between 0.1:9.9 and 9.9:0.1 added wherein the alkyl-or alkenyl-group is straight-or branched-chain and has an average number of carbon atoms of between 10 and 20.

3) Salts of alkyl or alkenyl sulfates wherein the alkyl or alkenyl group has an average number of carbon atoms of between 10 and 20.

4) Salts of olefinsulfonates having an average number of carbon atoms of between 10 and 20.

5) Salts of alkanesulfonates having an average number of carbon atoms of between 10 and 20.

6) Salts of a saturated or unsaturated fatty acid having an average number of carbon atoms of between 10 and 24.

7) Alkyl or alkenyl ether carboxylates having 0.5 to 8 moles per mole of ethylene oxide, propylene oxide, butylene oxide, ethylene oxide/propylene oxide in a ratio of between 0.1:9.9 and 9.9:0.1 or ethylene oxide/butylene oxide in a ratio of between 0.1:9.9 and 9.9:0.1 added wherein the alkyl or alkenyl group has an average number of carbon atoms of between 10 and 20.

8) Salts or esters of α-sulfofatty acids represented by the general formula:

$$R-\underset{\substack{|\\ SO_3Z}}{CH}CO_2Y$$

wherein Y stands for an alkyl group having 1 to 3 carbon atoms or a counter ion; Z stands for a counter ion and R stands for an alkyl or alkenyl group having 10 to 20 carbon atoms.

9) Amino acid type surfactants represented by the general formulas:

No. 1

$$R_1'-CO-\underset{\substack{|\\ R_2'}}{N}-\underset{\substack{|\\ R_3'}}{CH}-COOX$$

wherein R_1' stands for an alkyl or alkenyl group having 8 to 24 carbon atoms; R_2' stands for a hydrogen atom or an alkyl group having 1 to 2 carbon atoms; R_3' stands for an amino acid residue and X stands for an alkali or alkaline earth metal ion,

No. 2

$$R_1'-CO-\underset{\substack{|\\ R_2'}}{N}-(CH_2)_n-COOX$$

wherein R_1', R_2' and X are as defined above and n stands for a positive number of between 1 and 5,

No. 3

$$\begin{matrix} R_1' \\ R_1' \end{matrix} > N-(CH_2)_m-COOX$$

wherein R_1' is as defined above and m stands for a positive number of between 1 and 8,

No. 4

$$R_1'-\underset{\substack{|\\ R_4}}{N}-\underset{\substack{|\\ R_3'}}{CH}-COOX$$

wherein R_1', R_3' and X are as defined above and R_4 stands for a hydrogen atom, or an alkyl or hydroxyalkyl group having 1 to 2 carbon atoms,

No. 5

$$\begin{array}{l} R_5-\underset{\textstyle R_2'}{\underset{|}{N}}-\underset{\textstyle R_3'}{\underset{|}{CH}}-COOX \end{array}$$

wherein R_2', R_3' and X are as defined above and R_5 stands for a β-hydroxyalkyl or β-hydroxyalkenyl group having 6 to 28 carbon atoms, and

No. 6

$$\begin{array}{l} R_5 \\ \quad > N-\underset{\textstyle R_3'}{\underset{|}{CH}}-COOX \\ R_6 \end{array}$$

wherein R_3', R_5 and X are as defined above.

Among these surfactants, the surfactants 1), 2), 3) and 4) are preferred.

Furthermore, if necessary, other surfactants may be used, together with the above anionic surfactants, as an organic component to form a continuous phase. Examples of such surfactant include the following compounds:

10) Polyoxyethylene alkyl or alkenyl ethers wherein the polyoxyethylene moiety has added 1 to 20 mol of ethylene oxide and the alkyl or alkenyl group has an average number of carbon atoms of between 10 and 20.

11) Polyoxyethylene alkylphenyl ethers wherein the polyoxyethylene moiety has added 1 to 20 mol of ethylene oxide per mole, and the alkyl group has an average number of carbon atoms of between 6 and 12.

12) Polyoxypropylene alkyl or alkenyl ethers wherein the polyoxypropylene moiety has added 1 to 20 mole of propylene oxide per mole, and the alkyl or alkenyl group has an average number of carbon atoms of between 10 and 20.

13) Polyoxybutylene alkyl or alkenyl ethers werein the polyoxybutylene moiety has added 1 to 20 mol of butylene oxide per mole and the alkyl or alkenyl group has an average number of carbon atoms of between 10 and 20.

14) Nonionic surfactants having 1 to 30 total mol of ethylene oxide/propylene oxide or ethylene oxide/butylene oxide per mole added and an alkyl or alkenyl group of an average number of carbon atoms of between 10 and 20 (the ratio of ethylene oxide to propylene oxide or butylene oxide being between 0.1:9.9 and 9.9:0.1).

15) Higher fatty acid alkanolamides, or their alkylene oxide adducts, represented by the general formula:

$$R_{11}'\,CON < \begin{array}{l} (\overset{\textstyle R_{12}'}{\overset{|}{C}}HCH_2O)_{n3}H \\ (\underset{\textstyle R_{12}'}{\underset{|}{C}}HCH_2O)_{m3}H \end{array}$$

wherein R_{11}' stands for an alkyl or alkenyl group having between 10 and 20 carbon atoms; R_{12}' stands for a hydrogen atom or a methyl group; n^3 stands for an integer between 1 and 3 and m^3 stands for an integer between 0 and 3.

16) Esters of sucrose and a fatty acid having an average number of carbon atoms of between 10 and 20.

17) Monoglycerides of fatty acids wherein the fatty acid has an average number of carbon atoms of between 10 and 20.

18) Alkylamine oxides represented by the general formula:

$$R_{13}'-\overset{\textstyle R_{14}'}{\overset{|}{\underset{\textstyle R_{15}'}{\underset{|}{N}}}}\rightarrow O$$

wherein R_{13}' stands for an alkyl or alkenyl group having between 10 and 20 carbon atoms; R'_{14} and R_{15}' each stand for an alkyl group having between 1 and 3 carbon atoms.

19) Betaine type amphoteric surfactants represented by the general formulas:

No. 1

$$\begin{array}{c} \qquad\quad R_{22} \\ \qquad\quad | \\ R_{21} - N^{\ominus} - R_{23} - COO^{\ominus} \\ \qquad\quad | \\ \qquad\quad R_{22} \end{array}$$

wherein R_{21} stands for an alkyl, alkenyl, β-hydroxyalkyl or β-hydroxyalkenyl group having between 8 and 24 carbon atoms; R_{22} stands for an alkyl group having between 1 and 4 carbon atoms and R_{23} stands for an alkyl or hydroxyalkyl group having between 1 and 6 carbon atoms,

No. 2

$$\begin{array}{c} \qquad\qquad (C_2H_4O)_{n2}H \\ | \\ R_{21} - N^{\oplus} - R_{23} - COO^{\ominus} \\ | \\ \qquad\qquad (C_2H_4O)_{n2}H \end{array}$$

wherein R_{21} and R_{23} are as defined above and n^2 stands for an integer between 1 and 20, and

No. 3

$$\begin{array}{c} \qquad\quad R_{24} \\ \qquad\quad | \\ R_{21} - N^{\ominus} - R_{23}\,COO^{\ominus} \\ \qquad\quad | \\ \qquad\quad R_{24} \end{array}$$

wherein R_{21} and R_{23} are as defined above and R_{24} stands for a carboxyalkyl or hydroxyalkyl group having between 2 and 5 carbon atoms.

20) Sulfonic acid type of amphoteric surfactants represented by the general formulas:

No. 1

$$\begin{array}{c} \qquad\qquad\qquad\qquad R_{13} \\ \qquad\qquad\qquad\qquad | \\ R_{11}\,CONH - R_{12} - N^{\oplus} - R_{14} - SO_3^{\ominus} \\ \qquad\qquad\qquad\qquad | \\ \qquad\qquad\qquad\qquad R_{13} \end{array}$$

wherein R_{11} stands for an alkyl or alkenyl group having between 3 and 24 carbon atoms; R_{12} stands for an alkyl group having between 1 and 4 carbon atoms; R_{13} stands for an alkyl group having between 1 and 5 carbon atoms and R_{14} stands for an alkyl or hydroxyalkyl group having between 1 and 4 carbon atoms.

No. 2

$$\begin{array}{c} \qquad\quad R_{15} \\ \qquad\quad | \\ R_{11} - N^{\ominus} - R_{14} - SO_3^{\ominus} \\ \qquad\quad | \\ \qquad\quad R_{16} \end{array}$$

wherein R_{11} and R_{14} are as defined above and R_{15} and R_{16} each stand for an alkyl or alkenyl group having between 1 and 5 or between 8 and 24 carbon atoms, and

No. 3

$$\begin{array}{c} \qquad\qquad (C_2H_4O)_{n1}H \\ | \\ R_{11} - N^{\oplus} - R_{14} - SO_3^{\ominus} \\ | \\ \qquad\qquad (C_2H_4O)_{n1}H \end{array}$$

wherein R_{11} and R_{14} are as defined above and n_1 stands for an integer between 1 and 20

21) Phosphate surfactants e.g:

No. 1 Alkyl (or alkenyl) acid phosphates:

$$(R'O)_{n'}-\overset{\overset{\displaystyle O}{\|}}{P}-(OH)_{m'}$$

wherein R′ stands for an alkyl or alkenyl group having between 8 and 24 carbon atoms; n′ + m′ = 3 and n′ = 1 or 2.

No. 2 Alkyl (or alkenyl) phosphates

$$(R'O)_{n''}-\overset{\overset{\displaystyle O}{\|}}{P}-(OH)_{m''}$$

wherein R′ is as defined above; n″ + m″ = 3 and n″ = 1, 2 or 3.

No. 3 Salts of alkyl (or alkenyl) phosphates:

$$(R'O)_{n''}-\overset{\overset{\displaystyle O}{\|}}{P}-(OM')_{m''}$$

wherein R′, n″ and m″ are as defined above;

M′ stands for Na, K or Ca.

The amount of the organic component to be used is 10 to 75% by weight, preferably 15 to 60% by weight, of the weight of the detergent composition.

In the powdered detergent of the present invention, an inorganic component of the detergent is dispersed and enclosed in a continuous formed by an organic component of the detergent. The inorganic component contains a powdered alkali such as sodium carbonate, sodium sesquicarbonate, sodium silicate or the like as an essential ingredient. The organic component may contain a neutral salt such as Gluber's salt; a phosphate such as orthophosphates, pyrophosphates, tripolyphosphates, metaphosphates, hexametaphosphates or phytates or the following aluminosilicates:

No. 1 Crystalline aluminosilicates represented by the general formula:

$$x'(M'_2O \text{ or } M''O)\cdot A\ell_2O_3\cdot y'(SiO_2)\cdot w'(H_2O)$$

wherein M′ stands for an alkali metal; M″ stands for an alkaline earth metal which can be exchanged with calcium and x′, y′ and w′ stand for the number of moles of respective components and generally $0.7 \leqq x' \leqq 1.5$, $0.8 \leqq y' \leqq 6$ and w′ stands for a positive number.

No. 2 Aminosilicates, represented by the following formula, are preferred particularly as a builder:

$$Na_2O\cdot A\ell_2O_3\cdot nSiO_2\cdot wH_2O$$

wherein n is a number of between 1.8 and 3.0 and w is a number between 1 and 6.

No. 3 Amorphous aluminosilicates represented by the formula:

$$x(M_2O)\cdot A\ell_2O_3\cdot y(SiO_2)\cdot w(H_2O)$$

wherein M stands for Na and or K, and x, y and w stand for the number of moles of respective components and

$$0.7 \leqq x \leqq 1.2$$

$$1.6 \leqq y \leqq 2.8$$

and w stands for 0 or a positive number.

No. 4 Amorphous aluminosilicates represented by the following formula:

$$X(M_2O)\cdot A\ell_2O_3\cdot Y(SiO_2)\cdot Z(P_2O_5)\cdot \omega(H_2O)$$

wherein M stands for Na or K, and x, y, z and ω stand for the number of moles of respective components and

$$0.20 \leqq X \leqq 1.10$$

$0.20 \leqq Y \leqq 4.00$

$0.001 \leqq Z \leqq 0.80$

ω stands for 0 or a positive number.

These powdered alkalis preferably have a maximum particle size of at most 1500 μ, more preferably at most 1000 μ; and an average particle size of at most 500 μ, more preferably at most 200 μ. The amount of the inorganic component to be used is 10 to 75% by weight, preferably 15 to 60% by weight, of the total detergent weight.

In the high-density powdered detergent of the present invention, the primary or secondary particles of the inorganic component are dispersed in a matrix of the organic component. In some cases, however, part of the inorganic component can be added in a form not enclosed in the organic continuous phase.

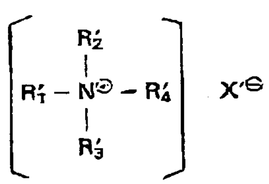
The weight to weight ration of the organic component, forming a continuous phase, to the inorganic component, enclosed in the continuous phase, is between 1/3 and 3/1, preferably between 1/2 and 2/1. If the ratio is less than 1/3, the organic component will not form a continuous phase, while if the ratio is more than 3/1, it will be difficult to pulverize the detergent. In the powdered detergent of the present invention, the inorganic component, particularly primary or secondary particles of powdered alkali, is coated with the organic component present as a continuous phase, particularly an anionic surfactant and the thickness of the coating is generally 10 to 500

The high-density powdered detergent of the present invention has a bulk density of at least 0.5 g/cm^3, preferably 0.5 to 1.5 g/cm^3, more preferably 0.5 to 1.2 g/cm^3 and most preferably 0.7 to 1.2 g/cm^3. If the bulk density is more than 1.5 g/cm^3, the powdered detergent will settle out in water as a result of a reduced solubility.

The following components may be added to the powdered detergent of the present invention.

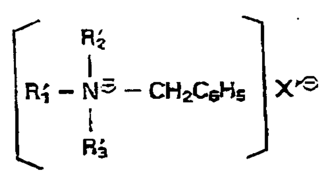
(1) Cationic surfactants represented by the general formulas:

No. 1



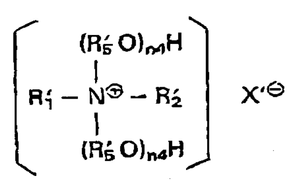
wherein at least one of R_1', R_2', R_3' and R_4' stands for an alkyl or alkenyl group having between 8 and 24 carbon atoms and the others stand for an alkyl group having between 1 and 5 carbon atoms,

No. 2



wherein R_1', R_2', R_3' and X' are as defined above, or

No. 3



wherein R_1', R_2' and X' are as defined above; R_5' stands for an alkylene group having between 2 and 3 carbon atoms and n_4 stands for a positive integer of between 1 and 20.

(2) Divalent metal ion scavengers

1) Salts of phosphonic acids, for example, ethane-1,1-diphosphonic acid, ethane-1,1,2-tri-phosphonic acid, ethane-1-hydroxy-1,1-diphosphonic acid and its derivatives, ethanehydroxy-1,1,2-triphosphonic acid, ethane-1,2-dicarboxy-1,2-diphosphonic acid or methanehydroxyphosphonic acid.

2) Salts of phosphono carboxylic acids, for example, 2-phosphonobutane-1,2-dicarboxylic acid, 1-phosphonobutane-2,3,4-tricarboxylic acid or α-methylphosphonosuccinic acid.

3) Salts of amino acids, for example, aspartic acid or glutamic acid.

4) Salts of aminopolyacetic acids, for example, nitrilotriacetic acid, ethylenediaminetetraacetic acid or diethylenetriaminetetraacetic acid.

5) Polyelectrolytes, for example, polyacrylic acid, polyaconitric acid, polyitaconic acid, polycitraconic acid, polyfumaric acid, polymaleic acid, polymethaconic acid, poly-α-hydroxyacrylic acid, polyvinylphosphonic acid, sulfonated polymaleic acid, maleic anhydride-diisobutylene copolymers, maleic anhydride-styrene copolymers, maleic anhydride-methyl vinyl ether copolymers, maleic anhydride-ethylene copolymers, maleic anhydride-ethylene crosslinked copolymers, maleic anhydride-vinyl acetate copolymers, maleic anhydride-acrylonitrile copolymers, maleic anhydride-acrylate copolymers, maleic anhydride-butadiene copolymers, maleic anhydride-isoprene copolymers, poly-β-ketocarboxylic acid derived from maleic anhydride and carbon monoxide, itaconic acid-ethylene copolymers, itaconic acid-aconitic acid copolymers, itaconic acid-maleic acid copolymers, itaconic acid-acrylic acid copolymers, malonic acid-methylene copolymers, methaconic acid-fumaric acid copolymers, ethylene glycolethylene terephthalate copolymers, vinylpyrrolidone-vinyl acetate copolymers, 1-butene-2,3,4-tricarboxylic acid-itaconic acid-acrylic acid copolymer, polyesterpolyaldehyde-carboxylic acid having quaternary ammonium groups, cis-isomer of epoxysuccinic acid, poly[N,N-bis-(carboxymethyl)acrylamide], poly(oxycarboxylic acid); succinate, maleate or terephthalate of starch, phosphates of starch, dicarboxystarch, dicarboxymethylstarch or succinates.

6) Non-dissociative polymers, for example, polyethylene glycol, polyvinyl alcohols, polyvinylpyrrolidone, chilled water-soluble urethanated polyvinyl alcohols.

7) Salts of organic acids, for example, diglycolic acid, oxydisuccinic acid, carboxymethyloxysuccinic acid, cyclopentane-1,2,3,4-tetracarboxylic acid, tetrahydrofuran-1,2,3,4-tetracarboxylic acid, tetrahydrofuran-2,2,5,5-tetracarboxylic acid, citric acid, lactic acid, tartaric acid; carboxylates of cane sugar, lactose, raffinose or the like; carboxymethylated pentaerythritol, carboxymethylated gluconic acid; condensates of polyhydric alcohols or sugars with maleic anhydride or succinic anhydride; condensates of oxycarboxylic acid with maleic anhydride or succinic anhydride; benzenepolycarboxylic acids such as mellitic acid; ethane-1,1,2,2-tetracarboxylic acid, ethene-1,1,2,2-tetracarboxylic acid, butane-1,2,3,4-tetracarboxylic acid, propane-1,2,3-tricarboxylic acid, butane-1,4-dicarboxylic acid, oxalic acid, sulfosuccinic acid, decane-1,10-dicarboxylic acid, sulfotricarballylic acid, sulfoitaconic acid, malic acid, oxydisuccinic acid, gluconic acid, CMOS or Builder-M.

(3) Anti-redeposition agents

One or more of the following compounds may be added to the detergent composition as an anti-redeposition agent in an amount of 0.1 to 5% wt. Examples of such agents include; polyethylene glycol, polyvinyl alcohol polyvinylpyrrolidone and carboxymethylcellulose.

(4) Bleaches eg:

Sodium percarbonate, sodium perborate, adducts of sodium sulfate with hydrogen peroxide, adducts of sodium chloride with hydrogen peroxide or the like.

(5) Enzymes (enzymes which can exhibit an inherent enzymatic action during washing)

Enzymes can be classified based on the reactions they catalyse into: hydrolase, hydrase, oxidoreductase, desmolase, transferase and isomerase, any of which can be used in the present invention. Particularly, hydrases are preferred and examples include proteases, esterases, carbohydrases and nucleases.

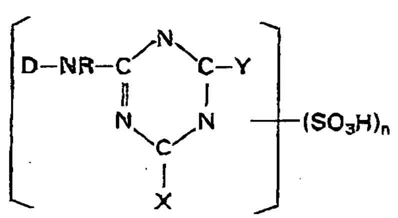
Examples of porteases include: pepsin, trypsin, chymotrypsin, collagenase, keratinase, elastase, subtilim, BPN, papain, bromelin, carboxypeptidase-A and -B, aminopeptidase, aspergillopeptidase A and B.

Examples of esterases include: gastric lipase, pancreatic lipase, plant lipase, phospholipase, choline esterase and phosphatase.

Examples of carbohydrase include: cellulase, maltase, saccharase, amylase, pectinase, lysozyme, α-glycosidase and β-glycosidase.

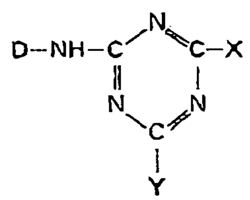
(6) Blueing agent

Various blueing agents may be added. Compounds having the following structures are preferred:



wherein D stands for a residue of monoazo-, disazo- or anthraquinone- type of blue or purple dyestuff; X and Y each stands for a hydroxy, amino, aliphatic amino group, which may be substituted with a hydroxy, sulfonic, carboxylic or alkoxy group; or an aromatic amino group which may be substituted with a halogen atom or a hydroxy, sulfonic, carboxylic, lower alkyl or lower alkoxy group; R stands for a hydrogen atom or a

lower alkyl group, except in the case where: R is a hydrogen atom and (1) X and Y simultaneously stand for a hydroxy or alkanolamine group or (2) one of X and Y is a hydroxy group and the other is an alkanolamine group; and n stands for a positive number of 2 or above.



wherein D stands for a residue of azo- or anthraquinone-type of blue or purple dyestuffs and X and Y are the same as or different to each other, and each stands for an alkanolamino residue or a hydroxy group.

(7) Caking inhibitor e.g:
p-Toluenesulfonates, xylenesulfonates, acetates, sulfosuccinates, talc, silica powder, clay, calcium silicate (for example, Microcel; a product of Johns Mansvills), magnesium oxide or the like.

(8) Antioxidant e.g:
t-Butylhydroxytoluene, 4,4'-butylidenebis-(6-t-butyl-3-methylphenol), 2,2-butylidenebis-(t-butyl-4-methylphenol), monostyrenated cresol, distyrenated cresol, monostyrenated phenol, distyrenated phenol, 1,1'-bis-(4-hydroxyphenyl)cyclohexane or the like.

(9) Fluorescent dye e.g.
4,4'-Bis-(2-sulfostyryl)-biphenyl salts, 4,4'-bis-(4-chloro-3-sulfostyryl)-biphenyl salts, 2-(styrylphenyl)naphthothiazole derivatives, 4'4'-bis(triazol-2-yl) stilbene derivatives, bis(triazinylamino) stilbenedisulfonic acid derivatives. One or more of the above compounds may be added to the powdered detergent in an amount of 0 to 1% by weight.

(10) Photo-active bleach, e.g:
One or more of sulfonated aluminum phthalocyanine and sulfonated zinc phthalocyanine can be added to the powdered detergent in an amount of 0 to 0.2% by weight.

(11) Perfume.
Then a process for preparation of the powder detergent composition, as disclosed above, will be illustrated below.
The powder detergent composition of the invention is prepared by the steps of: mixing and dispersing particles of an inorganic component with an organic component, in the melt state or dissolved state, removing liquid from the dispersion and pulverizing the dried dispersion to form a powder. In the process, a solution of the organic component, in an organic solvent, may be prepared in advance. In the step of mixing and dispersing an alkali powder with an anionic surfactant, they may be neutralized by one another. Preferably in the process, one or both, of a sulfonate and a sulfate, which have not yet been neutralized is used as the organic component, the neutralization, is completely or partially conducted, before the pulverisation step is conducted in advance to the pulverization step.
A preferred embodiment of the process will be explained below.
The process for the preparation of the high-density powdered detergent of the present invention is not particularly limited. Generally, the powdered detergent can be prepared by dispersing the inorganic component in the organic component, which is present as a melt or a solution. For example, the organic component is dissolved in an organic solvent, in which the inorganic component is insoluble. The inorganic component is dispersed in the solution, followed by the removal of the organic solvent. In particular, the following process, characterised by using an acid type of, that is to say, unneutralized anionic surfactant as the organic component in the compounding step is preferred. According to this process, the organic component containing an unneutralized anionic surfactant as a principal component, is mixed with the inorganic component containing a powdered alkali as a principal component, if necessary, together with a small amount of water. Thereby, the anionic surfactant is neutralized to form a continuous phase, while the inorganic component is dispersed and enclosed in the continuous phase, thus obtaining a mixed solid
The solid is granulated to a particle diameter of 40 to 2000 μ, preferably 125 to 1500 μ to obtain the desired powdered detergent. Furthermore in the above process, part of the final component of the detergent may be added during or after the solid granulating step, but in this case, the ratio of the solid mass to the final detergent composition is preferably at least 30%, more preferably at least 50% by weight. If the ratio is less than 30% by weight, it will be difficult to control the specific gravity of the powdered detergent.
Specifically, this invention provides a process for producing a high density granular detergent with improved solubility, which comprises a step of neutralizing an unneutralized sulfonation product and/or

sulfation product (hereinafter referred to as the un-neutralized product), a step of cooling and aging the un-neutralized product, that has been entirely or partially neutralized in the neutralizing step, and a step of granulating the same by pulverization or disintegration together with other detergent components.

The un-neutralized sulfonation or sulfation product usable in this invention can include, for example: linear or branched alkylbenzene sulfonic acid, having an alkyl group of average from 10 to 16 carbon atoms; an alkyl sulfate ester having an alkyl group of average from 10 to 18 carbon atoms; and an alkenyl ether sulfuric acid ester having an alkenyl group, in which from 0.5 to 8 mol on average of ethylene oxide, or a mixed alkylene oxide of ethylene oxide and propylene oxide or butylene oxide are added to one mole.

In practicing this invention, it is preferred to employ a combination system of the linear or branched alkylbenzene sulfonic acid and the alkyl sulfuric acid ester, because this will satisfy the required production cost and cleaning performance, as well as produce products easily pulverized and granulated in the subsequent steps. Furthermore, in practicing this invention, a combination comprising the un-neturalized product as one of the ingredients may be used, such as the combination of a sodium alkyl sulfate and a neutralized product of an alkyl benzene sulfonic acid.

For neutralizing the un-neutralized product used in this invention, an alkaline substance such as: sodium carbonate, sodium hydrogen carbonate and sodium silicate may be used; sodium carbonate being preferred in view of its reactivity, production cost, and availability of the raw materials, etc. The alkaline substance may preferably be used in an amount from 2 to 10 mol per mol of the un-neutralized product. While sodium carbonate may be used in the form either of dense ash (apparent specific gravity of about 1.0) or light ash (apparent specific gravity of about 0.6), since the reaction mainly occurs at the surface of sodium carbonate, better reactivity is obtained for the light ash due to its greater specific surface area. Pulverised sodium carbonate may also be used with improved reactivity. Furthermore, anhydrous or monohydrate sodium carbonate can also be used.

If sodium carbonate is used at less than 2 mol per one mol of the un-neutralized product the reaction will proceed, but over a longer timespan. On the other hand, although the reaction is possible if the amount of sodium carbonate exceeds 10 mol, it is preferably less than 10 mol in view of the aim of increasing the concentration of the active agent and from the production economics point of view. Water may be introduced as; the water content in other ingredients added upon neutralization; water content in sodium carbonate and water content in the un-neutralized product; or it may be added as additional water.

Other ingredients to be added upon neutralization basically comprise those capable of effectively behaving as detergent ingredients such as: an aqueous solution of sodium hydroxide; sodium silicate and an aqueous solution thereof; high molecular weight organic polymers containing carboxyl groups and/or hydroxy groups and aqueous solutions thereof; polyalkylene glycol and an aqueous solution thereof; perfumes and an aqueous solution thereof; fluorescent dyes and an aqueous solution thereof and pigment and an aqueous solution thereof. While a small amount of a builder such as zeolite, may be added upon neutralization, addition of a large amount is not desired since the structure of the zeolite is destroyed by the acid, reducing its ion exchange performance.

Some of the ingredients to be introduced have the effect of promoting the neutralizing reaction. For example, addition of a small amount of aqueous solution of sodium hydroxide or an aqueous solution of sodium silicate can promote the neutralizing reaction, as well as contribute to the reaction with the un-neutralized product as the alkaline ingredients. The sodium silicate used may include various types of sodium salts of silicic acid, for example: sodium methasilicate and hydrates thereof at various ratios, sodium orthosilicates and the like. Furthermore water glass, or the like, may also be used.

Furthermore, the high molecular weight organic polymer, containing carboxyl groups and/or hydroxy groups and an aqueous solution thereof, are effective as the detergent ingredient, and those having molecular weight from 500 to 100,000 are usually employed. By introducing these ingredients upon neutralization and mixing, a moderate hardness to the detergent may result, which may provide an advantageous effect in the subsequent steps of pulverization and granulation.

Suitable compounds as the high molecular weight organic polymer containing carboxyl groups and/or hydroxy groups can include: polymers and copolymers of acrylic acid, hydroxy acrylic acid, maleic acid, itaconic acid, mesaconic acid, aconitic acid and like other acids; as well as polyvinyl alcohol. Furthermore, polyalkylene glycols may include, for example, polyethylene glycol and polypropylene glycol.

Although the neutralizing reaction can be carried out with a water content of less than one mol per one mol of the un-neutralized product in this invention, the reaction rate is retarded requiring an increased retention time to complete the reaction. While on the other hand, although a water content in excess of 10 mole per one mole of the un-neutralized product may be used, it is undesirable since the adhesiveness of the neutralized product thus obtained is increased hindering the smooth pulverisation in the subsequent step. The preferred water content is within a range, preferably, from 1 to 10 mol and, more preferably, from 1 to 5 mol per one mol of the un-neutralized product.

The machine used in this invention for the neutralizing reaction may be, for example, a kneader that can provide an intense shearing force, for example, the Honda continuous kneader (manufactured by Honda Tekko K.K.), multipurpose continuous mixer (manufactured by Baker Perkins Inc.), KRC kneader (manufactured by Kurimoto Tekko K.K) or the Nesco kneader (manufactured by Fuji Sangyo K.K).

Each of the machines as described above has such a means of thoroughly mixing the un-neutralized product and the sodium carbonate or the like, and has a continuous and self-cleaning mechanism. Usually, a

retention time of about one minute within the device is sufficient.

Introduction of the un-neutralized product into the neutralizing device is carried out by a constant volume pump. Usually, linear or branched alkylbenzene sulfonic acid can be introduced from a store tank. While on the other hand, alkyl sulfuric acid ester, or alkenyl ether sulfuric acid ester, is fed to the neturalizing device directly after the sulfation, in order to prevent its decomposition during storage. While it is possible to temporarily store the alkyl sulfuric acid ester or the alkenyl ether sulfuric acid ester used, it is desired to restrict the retention time to within several hours, and keep the temperature above the melting point of the un-neutralized product but as low as possible.

The neutralizing reaction is started in the device as described above, having the self cleaning mechanism, and the reaction product is cooled and aged after the discharge from the machine and then transferred to the pulverizing step. The neutralization is completed in a neutralizing device or during the cooling and aging. In any case, it is sufficient that the neutralization is completed before the transfer to the pulverizing step.

The neutralizing reaction is preferably completed to 40-80%, usually in the neutralizing device, and the remaining neutralization is completed during cooling and aging. However, the reaction may be completed to 100 % in the neutralizing device. In the cooling and aging step, pores are produced in the neutralized product due to the degassing of gaseous carbon dioxide resulting from neutralization in the cooling and aging step and/or resulting from the neutralization step. The pores, formed by the generation of the gaseous carbon dioxide, have the effect of significantly improving the solubility of the final product, as well as enhancing its pulverisation properties. Furthermore, the cooling and aging can result in the crystallization of the neutralized product and the sodium carbonate or the like, etc. thereby reducing its shear strength, which is advantageous in the subsequent pulverisation steps. The retention time required for cooling and aging, while varying according to the degree of neutralization and the degree of crystallization sought is at least one minute preferably, 5 minutes and, more preferably, more than 10 minutes at less than 40°C, usually from 5 to 60 minutes being appropriate.

After the neutralization and aging steps, granular detergent containing more than 20 % by weight of the anionic surface active agent can be obtained. The cooling and aging may be carried out using, for example, a fluidizing bed, belt-type cooler, and cooling or, agitation under cooling, in a pneumatic transport machine, air slider or the like. By moulding the product, that has been entirely or partially neutralized into, a pellet-like configuration from 2 to 20 mm diameter and from 2 to 20 mm length, more efficient cooling procedures may be selected, and handling of the neutralized product such as transportation to the cooler and pulverizer may be facilitated.

Pellets with a diameter of less than 2mm, and less than 2 mm length are not practicable since it is difficult to uniformly mold them and they can easily disintergrate. Furthermore, those pellets exceeding 20 mm in diameter or 20 mm in length are not practicable since it is difficult to transport them to the cooler or pulverizer and supply them at a constant rate to the pulverizer. Accordingly, the size of the pellets when moulded from the un-neutralized product, which has been partially or entirely neutralized is preferably from 2 to 20 mm in diameter and from 2 to 20 mm in length and, preferably, from 5 to 10 mm in diameter, and from 5 to 15 mm in length. For moulding the un-neutralized product, which has been neutralized entirely or partially, into a pellet-like configuration, a conventional moulding machine, moulding granulator, or the like, may be used.

The temperature the neutralized product discharged from the neutralizing device is usually from 40°C to 100°C, which is reduced to less than 40°C by cooling. The neutralizing reaction is completed before the transfer to the pulverization step through cooling and aging. The cooled and aged neutralized product is pulverized with addition of other detergent ingredients. In this case, addition of powdered zeolite is desired since this enhances the pulverization properties and the powder properties of the final detergent product. Granular detergent with excellent flowability can thus be obtained by the above procedure.

The amount of the powdered zeolite added, is determined by the amount to be blended to the final product and is usually from 10 to 40 % by weight. Other detergent ingredients added upon pulverization can include, for example, carbonate such as calcium carbonate, sodium carbonate and sodium bicarbonate; sulfate such as sodium sulfate and magnesium sulfate; phosphate such as sodium tripolyphosphate, sodium pyrophosphate and sodium orthophospahte; as well as silicate such as sodium methasilicate and sodium orthosilicate.

Final detergent product can be obtained by mixing the pulverized detergent powder and other detergent ingredients. Other detergent ingredients can include, for example, nonionic surface active agent, foam stabilizer, textile softening agents, bleaching agents, enzymes, fluorescent dyes, pigments and perfumes.

The mixing can be carried out, for example, in a Schugi mixer, Lödige Mixer, P-K Blender, Pangranulator and fluidizing bed or the like. Introduction of the liquid ingredients, such as nonionic surface active agents, and perfumes can be carried out by spraying them into pulverized powder particles in the device.

In the case of a small amount of liquid ingredient, it may be introduced when the powder passes a spraying position on the conveyor belt. Furthermore, it is also possible to incorporate additives for the improvement of the powder's flow properties, such as a small amount of powdered zeolite.

The apparent specific gravity of the thus obtained powdered detergent, ranges from 0.5 to 1.0 g/cm^3 and usually from 0.6 to 0.98 g/cm^3, while varying depending on the starting material used, final blending composition, method of cooling and aging, and the granular size of the final detergent product.

The powdered detergent obtained by the spray drying method has an apparent specific gravity usually

from 0.25 to 0.4 g/cm^3, which is concentrated by about 2–3 times on the basis of volume. Furthermore, the thus obtained granular high density detergent has the advantage that it has a higher content of the active agent as compared with the powdered detergent produced by conventional spray drying methods.

While about 140 cc of conventional powdered detergents, obtained by spray drying methods is used per 30 liter of water for laundry the same degree of cleaning performance can be obtained with about 20 cc–50 cc of detergent powder produced according to this invention.

In this way, since the detergent obtained has reduced production cost, as well as packaging and transportation costs and in addition, since it has flow properties equivalent or superior to the powdered detergents obtained by the conventional spray drying method, it is very attractive to the consumer. Furthermore, with reference to its solubility, it has the same dissolution rate as the powdered detergent produced by the spray drying method ; is superior to the conventional product with respect to coagulating nature, and has excellent properties in use, coupled with high content of the anionic surface active agent and a high specific density.

Brief description of the drawings

Figures 1, 2 and *3* show a soft X-ray radiographs of the detergent of the present invention, the comparable detergent prepared by dry blending and a comparable detergent prepared by spray drying, respectively.

The present invention will be described in more detail by the following Examples, though the invention is by no means limited to them.

Example 1

Powdered detergents having the compositions shown in Table 1 were prepared and examined for powder structure, powder specific gravity and solubility. The results are shown in Table 2.

TABLE 1

Composition of detergent (The figures mean % by weight; hereinafter, the same applies)

Component	*Composition 1*	*Composition 2*	*Composition 3*
LAS	25	25	15
AS	10	10	10
AES	–	–	5
AOS	–	–	5
soap	1	1	1
nonion	3	3	3
zeolite	20	–	20
sodium tripolyphosphate	–	20	–
soda ash	25	25	25
sodium metasilicate	5	5	5
minor additives	3	3	3
volatile additives	6	6	6
Glauber's salt	balance	balance	balance

[NOTE]:
LAS: sodium linear alkylbenzene sulfonate ($C_{12}-C_{13}$)
AS: sodium alkylsulfate ($C_{14}-C_{15}$)
AES:sodium polyoxyethylenealkylsulfate ($C_{14}-C_{15}$, $\overline{EO} = 1.5$)
AOS: sodium α-olefinsulfonate ($C_{16}-C_{18}$)
nonion: polyoxyethylene alkyl ether ($C_{12}-C_{13}$, $\overline{EO} = 10$)

$\overline{EO}$: Average content of ethylene oxide/mol mol -1.

The process for the preparation of the detergent and the methods for the determination of the powdery structure, specific gravity and solubility of the detergent will be described.

1) Process for the preparation of detergent

(1) Process 1: In the following processes, an unneutralized, acid type of anionic surfactant was used as a starting material, but the composition of the detergent shown in Table 1 is the one of the final composition of the detergent.

The components of the composition 1 to 3 excluding zeolite or sodium tripolyphosphate were kneaded in a type of FM-NES-120 of Nesco kneader (a product of Fuji Industry Co., Ltd.) to thereby neutralize the anionic surfactant. Then, part (15%) of zeolite or sodium tripolyphosphate was added to the kneaded mixture and the resulting mixture was granulated with a hammer mill to a particle size of 1500 μm or less. The residue (5%) of the zeolite or sodium tripolyphosphate was added and the resulting mixture was further mixed with a Shugi-mixer to obtain a powdered detergent.

(2) Process 2: A slurry having the composition 1 to 3 (solid content: 60%) was prepared, dried with a drum drier and granulated with a screw extrusion granulator to a particle size of 1500 μ or below to obtain a powdered detergent.

(3) Process 3: A powdered surfactant having the composition 1 to 3 was prepared, and dry-blended with a Redige mixer to obtain a powdered detergent.

(4) Process 4: A slurry having the composition 1 to 3 (solid content: 60%) was prepared and spray-dried to obtain a powdered detergent.

2) Particle structure of powdered detergents was determined by soft X-ray radiographs.

3) Measurement of powder specific gravity was determined according to JIS K-3362.

4) Measurement of solubility 1.33 g of detergent was added to 1 ℓ of de-ionized water, kept at 10°C, under mild stirring by magnetic stirrer. After 3 minutes, the mixture was observed with the naked eye.

o: substantially all detergent was dissolved.

Δ: a little detergent was undissolved.

x: a considerable amount of detergent was undissolved.

TABLE 2

Experiment No.	*1 (Detergent of this invention)*	*2 (Comp. detergent)*	*3 (Comp. detergent)*	*4 (Comp. detergent)*	*5 (Detergent of this invention)*
Composition	1	1	1	1	2
Process	1	2	3	4	1
Particle structure of powdered detergent	particles comprising a continuous phase of the organic component and the inorganic component enclosed in the continuous phase	particle having a continuous phase of the inorganic component	organic component and inorganic component parti-	particle having a continuous phase of the inorganic component	the same particles. as the one described in Expt. 1.
Specific gravity (g/cm^3)	0.70	0.65	0.70	0.25	0.75
Solubility	o	x	x	o	o

TABLE 2 (cont'd)

Experiment No.	6 *(Comp. detergent)*	7 *(Comp. detergent)*	8 *(Detergent of this invention)*	9 *(Comp. detergent)*	10 *(Comp. detergent)*
Composition	2	2	3	3	3
Process	2	3	1	2	3
Particle structure of powdered detergent	the same particle as those described in Expt. 2	the same particle as those described in Expt. 3	the same particle as those described in Expt. 1	the same particle as those described in Expt. 2	the same particle as those described in Expt. 3
Specific gravity (g/cm^3)	0.68	0.71	0.65	0.60	0.63
Solubility	×	×	o	×	×

Only detergents of Experiment Nos. 1, 5 and 8 according to the present invention, which were prepared by Process 1 and comprising particles with a continuous phase of the organic component and an inorganic component enclosed in the continuous phase, exhibited a high density and a high solubility.

Example 2

The organic component and inorganic component shown in Table 3 were fed to a continuous kneader (a product of Honda Ironworks Co., Ltd.) to carry out neutralizaton and kneading. Part (15%) of zeolite was added to the kneaded mixture and the resulting mixture was granulated with a paraplex mill (a product of fuji Industry Co., Ltd.) to a particle size of 1500 μm or less. The residue of zeolite, minor additives and Glauber's salt were added to the mixture. The resulting mixture was further mixed with a rotary mixer to obtain a powdered detergent. The powdered detergent was examined for specific gravity, fluidity and solubility. The results are shown in Table 3. It should be understood from the results that a high-density powdered detergent, having an improved fluidity and a high solubility, can be obtained only when the ratio the organic component to the inorganic component is between 1:3 and 3:1.

TABLE 3

		Experimental No. 1	2	3	4	5
Organic component	LAS (Note)	8	11.5	17.5	23.5	26
	AS	8	11.5	17.5	23.5	26
Inorganic component	soda ash	39	35	26	17	14
	sodium meta-silicate	13	12	9	6	4
zeolite		20	20	20	20	20
minor additives		3	3	3	3	3
volatile		5	5	5	5	5
Glauber's salt		balance	balance	balance	balance	balance
organic component/ inorganic component		1/3	1/2	1/1	2/1	3/1
specific gravity (g/cm^3)		0.83	0.75	0.70	0.68	0.65
fluidity (second)		8.6	8.4	8.5	9.0	11.0
solubility		Δ	o	o	o	o

Note) LAS and AS are the same as the one described in Example 1.

In preparing the above detergent, an unneutralized acid type of anionic surfactant was used, but the components shown in Table 3 are those present in the final detergent composition.

The process for the measurement of specific gravity, fluidity and solubility will now be described.

1) Measurement of specific gravity: the same process as the one described in Example 1

2) Fluidity: the time required for 100 cc of a powdered detergent to fall through a gravimeter for powder according to JIS K-3326.

3) Solubility: the same process as the one described in Example 1

The high-density powdered detergent of the present invention can be distinguished from those of the prior art by; the difference in particle shape which is recognized by microscopic examination and, by the difference in the distribution of the inorganic component which is recognized by soft X-ray radiographic examination.

Figures 1 to 3 show soft X-ray radiographs (x 150) of the detergent of the present invention and soft X-ray radiographs of comparative detergents, respectively.

Figure 1 shows a particle of the detergent according to the present invention. The particle diameter of the detergent of the present invention (before the later addition of the inorganic component, determined by microscopic examination, is larger than that of the inorganic component, determined by soft X-ray radiographic examination, which implies that the organic component form a continuous phase and the phase encloses primary or secondary particles of the inorganic component.

When an inorganic component such as zeolite is added later to the detergent of the present invention, the detergent obtained is shown by micrographic examination to consist of particles covered with zeolite, and it is shown by by soft X-ray radiographic examination that the inorganic component is present all over the particle.

Figure 2 shows a particle of the detergent prepared by dry blending according to Process 3. The particle size (shape) of the detergent, determined by micrographic examination, is nearly equal to that of the inorganic component, determined by soft X-ray radiographic examination, and particles which can not be observed by soft X-ray radiographic examination are present. That is to say, it is evident that particles of the organic component and particles of the inorganic component are present separately.

Figure 3 shows a particle of the detergent prepared by spray drying according to Process 3. The shape of the particle determined by microscopic examination is nearly the same as the one determined by soft X-ray radiographic examination, which implies that the outer shell of the particle is comprised of the inorganic component and that the organic component is present within the particles.

Example 3

To a FM-NES-120 type Nesco kneader (manufactured by Fuji Sankyo K.K.), was supplied at a constant rate the starting material of the following composition:

Linear alkylbenzene sulfonic acid (average carbon atom number in alkyl group C = 12)	45 kg/H (132.5 mol)
Alkyl sulfuric acid ester (C = 12.5)	15 kg/H (56.4 mol)
Anhydrous light ash	45 kg/H (424.5 mol)
20 % aqueous solution of NaOH	5 kg/H (NaOH 25.0 mol) (H_2O 222.2 mol)

The linear alkylbenzene sufonic acid used here was stored at about 30°C after sulfation by a conventional process and it had a purity of 96 %, free sulfuric acid content of 1.2 %, water content of 0.4 % and oil content of 2.4 %. Furthermore, the alkyl sulfuric acid ester was used directly in this example after sulfation by a known process.

The average retention time in the kneader was about 1 min. and the temperature of the composition at the exit was about 70°C. It was cooled to about 30°C in a belt type cooling device. The cooling time was 30 min and it was further retained for about 10 min after it had been cooled to less than 40°C. The neutralization was complete. After the cooling, the following composition was obtained.

Sodum salt of linear alkylbenzene sulfonate	46.0 kg/H
Alkyl sufuric acid ester	16.0 kg/H
Sodium carbonate	35 kg/H
Oil and sodium sulfate	2 kg/H
Water	6 kg/H

Powdered zeolite (Toyobuilder, powdery product manufactured by Toyo Soda K.K.) was mixed in at 20 parts by weight to 80 parts by weight of the above-mentioned composition, and then pulverized to less than 1000 um size by a hammer mill. The pulverization resulted in a powdered detergent with good flow properties and low coagulating properties. The powdered detergent thus obtained has an apparent specific gravity of 0.7 g/cc. Evaluation was carried out on the powered detergent thus obtained with respect to cleaning performance, foaming performance, solubility, coagulating property or the like.

The results of the evaluation are shown in Table 4. The cleaning performance was identical with the commercialized detergent (25 % by weight of surface active agent) for the amount of the active agent used, and other powder properties were also the same.

Example 4

The composition prepared in the same manner as in Example 3 at an exit temperature of about 70°C was moulded into pellets of 5 mm diameter and about 10 mm length by using a EXD 60 type Pelleter Double (manufactured by Fuji Powdery K.K) and a cutting device. Then, these were cooled using a fluidizing bed to about 50°C. Cooling time was 15 min and they were maintained for about 10 min after cooling to 40°C. The neutralization was complete. The following composition was obtained after the cooling.

Sodium salt of linear alkylbenzene sulfonate	46.0 kg/H
Alkyl sufuric acid ester	16.0 kg/H
Sodium carbonate	35 kg/H
Oil and sodium sulfate	2 kg/H
Water	6 kg/H

Powdered zeolite (Toyobuilder, powdery product manufactured by Toyo Soda K.K.) was mixed in at 20 parts by weight to 80 parts by weight, of the above-mentioned composition and then pulverized to less than 1000 um size by a hammer mill. The pulverization resulted in a powdered detergent with good flow properties and low coagulating properties. The powdered detergent thus obtained has an apparent specific gravity of 0.7 g/cm^3. Evaluation was carried out on the powder thus obtained with respect to cleaning performance, foaming performance, solubility, coagulating property or the like.

The results of the evaluation are shown in Table 4. The cleaning performance was identical with the commercialized detergent (25 % by weight of surface active agent) for the amount of active agent used, and other powder properties were also the same.

The method of evaluating the detergent in Table 4 is as below :

(I) Detergent performance

The detergent performance was measured according to the method of JIS by a Shaffers pair comparative method based on the commercialized detergent.

5% degree of freedom + superior

± equal

− inferior

(II) Foaming power

30 liter tap water at 20°C were poured into a commercial electrical washing machine, to which were added and dissolved 20 g of the detergent, according to this invention (40 g of the commercial detergent as a control). 1 Kg of cotton under wear and artificial oil contamination (cotton seed oil) were placed into the machine and the foaming state was judged after 10 minutes' heavy agitation.

(III) Solubility

1.33 g of the detergent were added to one liter of deionized water kept at 10°C and slowly stirred by a magnetic stirrer. 5 minutes later, the detergent liquid was passed through a 200 mesh sieve and the weight of the residues on the sieve was determined.

(IV) Coagulating property

Detergent powder was sealed in a commercial detergent carton which had not been treated with a moisture proofing agent. 2.6 kg of the product according to this invention was sealed in such a carton of 22 cm (W) × 30.5 cm (h) × 6.5 cm (d) 2.6 kg of the control product was sealed in a carton of 25.5 × 37.5 × 10. After standing at 30°C and 80 % RH (relative humidity) for three weeks, the powder was discharged on to a 4mm × 4 mm sieve and the weight of the powder A (kg) remained on the sieve and the weight of the powder B (g) not retained on the sieve were measured and the passage ratio (c) was determined according to the following equation:

$$C\,(\%) = \frac{B}{A+B} \times 100$$

TABLE 4

	cleaning performance	*Foaming power (maximum foam height cm)*	*Solubility (%)*	*Coagulating property C (%)*
Example 3	±	10	0.1	65
Example 4	±	10	0.2	60
Commercial detergent	standard	10	0.8	58

CLAIMS

1. A powder detergent composition having a high density, which comprises an organic component a part or all of which forms a continuous phase and particles of an inorganic component dispersed in the continuous organic component.

2. A powder detergent composition as claimed in Claim 1, in which the organic component is an anionic surfactant and the inorganic component is a powdered alkali.

3. A powder detergent composition as claimed in Claim 1 or Claim 2, in which a weight ratio of the organic component to the inorganic component ranges from 1/3 to 3/1.

4. A powder detergent composition as claimed in any preceding claim which has a specific gravity of 0.5 g/cm^3 or larger.

5. A process for preparing a powder detergent composition having a high density, which comprises the steps of mixing and dispersing particles of an inorganic component in an organic component in the form of a melt or a solution, drying the resulting dispersion and pulverising the dried dispersion.

6. A process as claimed in claim 5, which includes the preliminary step of preparing a solution of the organic component in an organic solvent.

7. A process as claimed in claim 5, or claim 6, which comprises the step of mixing and dispersing a powdered alkali with an anionic surfactant so that they may neutralize one another.

8. A process as claimed in any of claims 5 to 7 in which a sulfonate or a sulfate or a mixture thereof which has not yet been neutralized is used as the organic component, neutralization is completely or partially conducted, and a cooling and aging step is conducted prior to the pulverization step.

9. A process as claimed in any of claims 5 to 8 in which the pulverization step is effected with incorporation of another detergent component.

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				DE	3528190	A	19850806
DE	3528190	A1	19860213	JP	16460984	A	19840806
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